U.S. Environmental Protection Agency

Environmental Technology Verification Program

For Metal Finishing Pollution Prevention Technologies

Verification Test Plan

Evaluation of USFilter Membralox® Silverback™ Model 900 Alkaline Cleaner Recycling System

Revision 0

April 4, 2000

Concurrent Technologies Corporation is the Verification Partner for the EPA ETV Metal Finishing Pollution Prevention Technologies Pilot under EPA Cooperative Agreement No. CR826492-01-0.

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Alkaline Cleaner Recycling System

April 4, 2000

Prepared by: ETV-MF Program and
USFilter Corporation

TITLE: **EVALUATION OF USFILTER MEMBRALOXÒ SILVERBACKÔ MODEL 900 ALKALINE CLEANER RECYCLING SYSTEMS**

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Environmental Technology Verification Program for Metal Finishing Pollution Prevention Technologies (ETV-MF) Verification Test Plan for the Evaluation of USFilter Membralox® Silverback™ Model 900 Alkaline Cleaner Recycling System.

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1.0 INTRODUCTION

The purpose of this test plan is to document the objectives, procedures, equipment, and other aspects of testing that will be utilized during verification testing of the Membralox® Silverback™ Model 900 alkaline cleaner recycling system. This test plan has been prepared in conjunction with the U.S. Environmental Protection Agency's (EPA's) Environmental Technology Verification Program for Metal Finishing Pollution Prevention Technologies (ETV-MF). The objective of this program is to identify promising and innovative pollution prevention technologies through EPA supported performance verifications. The results of the verification test will be documented in a verification report that will provide objective performance data to metal finishers, environmental permitting agencies, and industry consultants. A verification statement, which is an executive summary of the verification report, will be prepared and signed by the EPA National Risk Management Research Laboratory Director.

Alkaline cleaning baths are used by nearly all metal finishing companies to remove oil and other soils prior to electroplating, anodizing, painting, or other finishing processes. These processes are very sensitive to the presence of surface contaminants. Usually, alkaline cleaning baths are used until they become moderately contaminated with oil and suspended solids, and then they are discarded and replaced with fresh cleaning chemicals, despite the fact that a significant portion of the bath's constituents are still usable. If used beyond a certain point, the contamination will prevent good surface cleaning, which may lead to defects in a part's finish. Also, contaminants in the cleaning baths can carry over to subsequent tanks and cause deterioration of those solutions. The practice of discarding and replacing contaminated cleaning baths is expensive due to the cost of cleaning chemistry (most cleaners are proprietary formulations) and the cost of disposal.

This project will evaluate the ability of the Silverback™ unit to separate oil and suspended solids from the alkaline cleaning bath under different soil loading rates. The Silverback™ unit employs a ceramic microfiltration membrane to perform the separation of contaminants and cleaning solution.

Testing of the Silverback™ unit will be conducted at Gates Rubber Company located in Versailles, MO. The Gates Rubber Company manufactures a wide range of hydraulic couplings and hose assemblies. Alkaline cleaning is performed on metal parts at strategic times during the manufacturing process, including prior to zinc electroplating. Used alkaline cleaning solution is piped to a storage tank that feeds the Silverback™ unit. Recovered alkaline cleaner from the unit is pumped to a recycled cleaner tank and reused.

Testing will be conducted over a two week time period, which will be divided into two 1-week test runs. During the first test run, the unit will be operated at the normal soil-loading rate. During the second test run, the soil-loading rate will be doubled. During both test runs, the process will be monitored to measure the soil-loading rate, the recovery efficiency of the unit, and certain process measurements.

The test plan described in this document has been structured based on a format developed for ETV-MF projects. This document describes the intended approach and explains testing plans with respect to areas such as test methodology, procedures, parameters, and instrumentation. Also included is Quality Assurance/Quality Control requirements of this task that will ensure the accuracy of data, data interpretation procedures, and worker health and safety considerations.

2.0 TECHNOLOGY DESCRIPTION

2.1. Theory of Operation

Alkaline cleaning solutions are a mixture of chemicals, including surfactants, alkali salts, caustic soda, phosphates, and complexing agents. These baths build up concentrations of oil and solids during use. Free oils can be removed by simple skimming, and most solids can be removed by settling and/or cartridge filtration. However, emulsified oils and colloidal solids are not affected by these devices. At some point, the cleaning efficiency of the bath is impaired and the solution is discarded, despite the fact that most of the bath's constituents are still usable. In many cases, heavy-duty cleaners must be replaced once per week. The microfiltration technology separates the emulsified oils and colloidal solids from the aqueous cleaning solution, thereby extending the life of the bath. This technology is also applicable to the recovery of cleaning solution drag-out from rinse waters.

Many commercial microfiltration systems used for this application, including the Silverback™ technology, employ ceramic filter membranes in a crossflow filtration configuration. These membranes are a relatively new development that permits application of microfiltration to solutions and emulsions that are both heated and corrosive. The ceramic membranes are produced in a range of pore sizes that selectively permit a large percentage of the surfactants to pass through the membrane. A typical pore size is 0.8 microns (µ) and most microfiltration units have pore sizes greater than 0.2 µ.[1] Crossflow filtration, as opposed to barrier or "dead-end" filtration, permits the application of this technology to high solids-feed streams. With dead-end filtration, all of the feed solution is forced through filtration media by an applied pressure. With a high solids-feed stream, the pores of a dead-end filtration device plug. With crossflow filtration, the fluid to be filtered is pumped across the membrane, parallel to its surface. By maintaining a high velocity across the membrane, the retained material is swept off the membrane surface. [Ref. 1, 2]

2.2 Description of Membralox® Silverback™ Unit

The Membralox® Silverback™ Model 900 is an alkaline cleaner recycling system. This unit contains a Membralox® 7P19-40 module made up of seven alpha alumina elements, each with 19 lumens (channels) that are 4.0 mm in diameter. The inside of each channel is lined with an alpha alumina or zicronia membrane having a total surface area of 1.68 m^2 (18.1 ft^2) and a pore size of 0.2 µ. The manufacturer

[1] 1 millimeter = 1,000 microns

indicates that the membrane is impervious to nearly all chemicals except for phosphoric and hydrofluoric acids and can tolerate temperatures up to 200^{o}F.

A diagram of the Silverback™ Model 900 is shown in ***Figure 1***. In operation, the contaminated cleaner enters a two-compartment 304 stainless steel tank through a filter (polypropylene sock and stainless steel basket) that removes large particulate material from the feed stream. The level in the tank is maintained by a level switch, which controls the tank inlet valve and also acts as a low-level cutoff for the system pump. The oils accumulate in the initial compartment (referred to as the settling tank) and can be removed on a periodic basis through a drain port located on the upper part of the tank.[2] The liquid then moves to a second tank compartment through a sub-surface passage; thereby leaving the floating oils in the first compartment. The liquid in the second compartment (referred to as the recirculation tank) is pumped through the ceramic filter located in the microfiltration module. A portion of the water and cleaner chemicals are forced through the ceramic membrane, while the oil and other soils are retained and recycled back to the tank. A range of system capacities is available for feed stream flow rates of 150 gpd to 3,900 gpd (model 900 has a design capacity of 900 gpd). According to the manufacturer, the flux rate of the membrane, which is dependent on the composition and viscosity of the alkaline cleaner feed, is approximately 50 to 100 gallons per square foot per day (GFD).

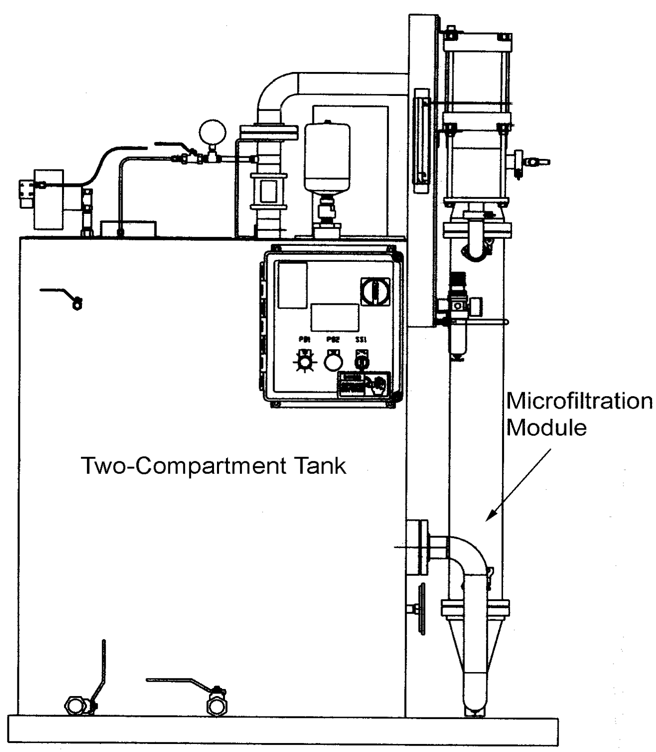


Figure 1. Diagram of the Membralox® Silverback™ Technology

[2] This drain is not used at the Gates Rubber. No floating oils are removed.

The Membralox® Silverback™ Model 900 is equipped with a backpulsing device that is designed to clean the filtration modules during operation by periodically pushing solution under pressure against the normal filtration direction. The frequency and duration of the backpulse cycle is timer activated and controlled. During the backpulse, a volume of approximately 700 ml of permeate is pushed back through the ceramic membranes in about one tenth of a second. The unit then returns to its normal flow pattern.

2.3 Commercial Status

The Membralox® Silverback™ Technology is fully commercialized. Approximately 500 units have been sold.

2.4 Pollution Prevention Classification

The Membralox® Silverback™ Model 900 is a bath maintenance technology. Bath maintenance refers to a range of pollution prevention practices and technologies that preserve or restore the operating integrity of metal finishing process solutions, thereby extending their useful lives. Due to rising costs for chemicals, energy, and treatment/disposal and increasingly more stringent environmental requirements, bath maintenance has become a greater priority to metal finishing companies, and the methods and technologies they employ have increased in sophistication. Today, firms are willing to expend significant amounts of capital and operating funds for equipment and methods that primarily reduce the disposal frequency of their baths. In addition to extending bath lives, solution maintenance often improves the average operating efficiency and effectiveness of a process solution and therefore has a positive impact on production rates and finish quality.

2.5 Environmental Significance

The Membralox® Silverback™ Model 900 is employed to reduce the disposal frequency of alkaline cleaning baths. Alkaline cleaning baths are used by nearly all metal finishing companies to remove oil and other soils prior to electroplating, anodizing, painting, or other finishing processes. These processes are very sensitive to presence of surface contaminants. Usually, alkaline cleaning baths are used until they become moderately contaminated with oil and suspended solids, and then they are discarded and replaced with fresh cleaning chemicals. If used beyond a certain point, the contamination will prevent good surface cleaning, which may lead to defects in a part's finish. Also, contaminants in the cleaning baths can carry over to subsequent tanks and cause deterioration of those solutions. Spent cleaning baths can be hauled to a treatment, storage, and disposal (TSD) facility or treated on-site. The off-site treatment/disposal option is typically expensive, and on-site treatment can present difficulties due to the presence of chelating compounds found in most cleaners. These compounds tend to prevent complete precipitation of heavy metals and therefore may interfere with a company's ability to meet effluent guidelines. Also, treatment of cleaning baths generates significant quantities of sludge.

The disposal frequency of cleaning baths varies from company to company and depends on several factors, including type of cleaner, type of soil, soil loading rate, and cleanliness requirements. Prior to implementing a bath maintenance technology, Gates Rubber Company disposed of their cleaning baths (approximately 2,500 gal.) approximately every two weeks. Subsequent to implementation of the technology, the disposal frequency has been reduced to every six months. Additionally, the contents of the Silverback™ recirculation tank are discarded every week (100 gal.). Records from Gates Rubber Company will be reviewed during testing and summarized in the verification report as background information. These records are extensive and include a time period prior to installation of alkaline cleaner recycling equipment. The verification report will include a statement clearly indicating that these data are present for the purpose of providing background information and have not been verified.

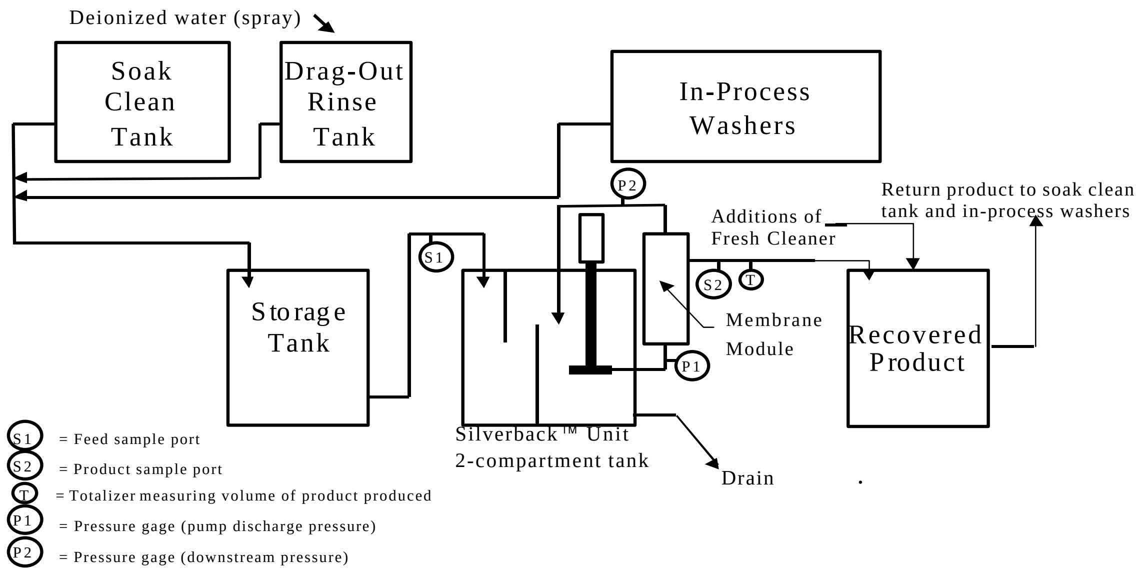
2.6 Local Installation

The Gates Rubber Company located in Versailles, MO, was selected by USFilter as the test site for the Membralox® Silverback™ Model 900. The unit that will be evaluated during this ETV-MF project was installed in September 1999. However, Gates Rubber has previous experience with the Membralox® Silverback™, dating back to 1997. The Gates Rubber Company manufactures a wide range of hydraulic couplings and hose assemblies. They have an 115,000 ft^2 manufacturing facility. Alkaline cleaning is performed at various points in the plant. There are 12 in-process cleaning tanks present in areas such as machining. Eleven of these units hold 40 gal. of alkaline cleaner and one holds 75 gal. The largest cleaning operation is located on the barrel plating (zinc) line, where there is a 1,800 gal. soak cleaning tank and a 1,800 gal. electrocleaning tank. The 12 in-process cleaning tanks and the soak cleaning tank are plumbed into the cleaner recycling system that will be tested during this project. The electroclean tank is serviced by a separate recycling system and will not be a part of this effort.

The predominant oil found on the parts processed through the cleaning systems is a cutting oil (Tru Cut 2582). The MSDS for this product indicates it is a petroleum hydrocarbon with additives. Its hazardous components are hydrotreated light napthenic oils and hydrotreated heavy napthenic oils. Laboratory analyses show that it is a mixed base mineral oil that contains an ester-based additive. Other additives include a phenolic anti-oxidant. Traces of cellulose or a derivative thereof are also present.

A diagram of the closed-loop cleaner recycling system involved in this ETV-MF project is shown in ***Figure 2***. The solution that is processed by the Silverback™ unit comes from three sources, including the soak clean tank and drag-out rinse tank on the zinc plating line and from in-process washers located in various parts of the manufacturing facility. Each of these three sources is connected by piping (represented by lines with arrows in Figure 2) to the storage tank.

Figure 2. Alkaline Cleaner Recycling at Gates Rubber Company



The solution conveyed from in-process cleaning and the soak clean tank is used alkaline cleaner. The solution conveyed from the drag-out rinse tank to the storage tank is rinse water that contains alkaline cleaner. De-ionized (DI) water is added to the drag-out rinse tank from spray bars located above the drag-out rinse tank. The addition of this water causes the solution in this tank to overflow a weir and the overflow is conveyed to the storage tank. The DI water sprays are automatically activated when a barrel is removed from the drag-out rinse tank.

The solution that is in the storage tank is pumped to the Silverback™ unit and is processed at a rate of approximately 1.1 gpm. The recovered product from the Silverback™ unit is pumped to a recovered product tank, from where it is drawn-off and reused in the soak clean tank and in-process washers.

Barrels exiting the drag-out rinse tank are further processed in an electro-cleaning tank and subsequently zinc plated. The electro-cleaning tank is not connected to the recovery system being tested during this project.

The alkaline cleaner used at this facility is CLEAN-R-120GR, which is formulated and sold by PAVCO, a company located in Cleveland, OH. The MSDS for this product can be found in Appendix A. The concentration of the cleaner is controlled, based on alkalinity concentration. The product is purchased as a single component concentrated liquid. When measurements indicate that the alkalinity concentration is below a recommended operating level, additional liquid concentrate is added to the cleaning system.

PAVCO indicated that the cleaner is composed primarily of the following chemicals or classes of chemicals: sodium hydroxide, phosphate, glycol ether, octylphenol, and amine.

3.0 EXPERIMENTAL DESIGN

3.1 Test Goals and Objectives

The overall goal of this ETV-MF project is to evaluate the ability of the Silverback™ unit to recover alkaline cleaning chemistry that is used under actual production conditions and to evaluate and characterize the operation of the unit through measurement of various process parameters.

The following is a summary of primary project objectives. Under normal system operating setpoints for the installation at Gates Rubber Company and varying contaminant-loading rates:

- Prepare a material balance for alkaline cleaner constituents and soils in order to:
    1. Evaluate the ability of the Silverback™ unit to recover alkaline cleaner.
    2. Evaluate the ability of the Silverback™ unit to remove contaminants from used cleaning solution.
- Determine the cost of operating the alkaline cleaning recovery system for the specific conditions encountered during testing.
    1. Determining labor requirements needed to operate and maintain the Silverback™ unit.

2. Determining the quantity of energy consumed by the Silverback™ unit during operation.

- Quantify the environmental benefit by determining the reduction in bath disposal frequency.

3.2 Critical and Non-Critical Measurements

Measurements that will be taken during testing are classified below as either critical or non-critical. Critical measurements are those that are necessary to achieve the primary project objectives. Non-critical measurements are those related to process control or general background readings.

Critical Measurements:

- solution processing rate (gallons per minute)
- chemical characteristics of feed and product solutions (alkalinity, phosphate, glycol, total phenol, total kjeldahl nitrogen (TKN), ammonia, oil and grease (oil), total solids, and total suspended solids (TSS))
- concentrated cleaning solution additions (gallons) and related costs
- chemical characteristics of concentrated cleaning solution (alkalinity, phosphate, glycol, total phenol, total kjeldahl nitrogen (TKN), ammonia, oil and grease (oil), total solids, and total suspended solids (TSS))
- waste volumes (gallons) and chemical characteristics (alkalinity, phosphate, glycol ether, octylphenol, oil , total solids, and TSS) of those wastes
- chemical characteristics of feed and product solutions (alkaline cleaner components and contaminants)
- cleaning chemical additions (gallons) and related costs
- O&M labor requirements (hours) and costs
- production throughput measured in terms of square footage of metal parts cleaned
- energy use for pumps (kWh) and reheating of recycled solution (BTUs) and costs

Non-Critical Measurements:

- solution temperature (degrees Fahrenheit) in second compartment (recirculation tank) of Silverback™ unit
- differential pressure (psig) across the membrane (pump discharge pressure/downstream pressure)
- backpulse frequency (minutes) and duration (seconds) settings

3.3 Test Matrix

Testing will be conducted in two distinct test periods, with each test period having a duration of approximately one week. The soil loading of the unit will be varied from the first test period to the second by a factor of approximately two.

During the first test period, the entire cleaning line and alkaline recycling system will be operated using normal operating conditions found at Gates Rubber Company (see section 2.6).

Therefore, during the first test period, the soil concentration in the Silverback™ unit feed solution will be at a "normal" level. This "normal" level will be quantitatively determined during the first test period by collecting and analyzing samples of the feed solution.
At the completion of the first test period, the contents of the two-compartment tank of the Silverback™ unit, which holds the soil removed from the alkaline cleaning solution, will be removed from the Silverback™ unit.

During the second test period, the entire cleaning line and alkaline recycling system will be operated using the normal operating procedures (see section 2.6), with one exception. To evaluate the operation of the Silverback™ unit under a high soil loading condition than is normally found at Gates Rubber Company, the soil that was removed during the first test period from the Silverback™ unit will be bled into the feed solution (bled at a uniform rate into the storage tank) during the entire second test period. These procedures will approximately double the soil-loading rate during the second test period. The actual soil-loading rate during the second test period will be quantitatively determined by collecting and analyzing samples of the feed solution.

The higher soil loading during the second test period may cause operational problems, such as plugging the membrane. This could occur, for example, due to a buildup of solids in the recirculation tank. Efforts will be made to conduct the second test for a minimum of five days. Any maintenance requirements (e.g., cleaning of filter) or operational changes needed to keep the unit operating will be conducted and recorded. However, if it is necessary to discharge the solution in the recirculation tank (e.g., due to solids buildup) prior to operating for five days, then the test will be considered complete. Any data collected during a shortened test period will be considered usable in the verification report subject to data quality requirements (see section. 4). The cause of the shortened test period will be noted and discussed in the report.

Test objectives and measurements are summarized in ***Table 1***.

3.4 Testing And Operating Procedures

3.4.1 Set-Up and System Initialization Procedures

Prior to startup, the recirculation tank will be drained and cleaned and the membrane will be cleaned (see Appendix B, Silverback™ O&M manual for procedural details). The recirculation tank will then be filled with cleaner solution from the feed tank and the Silverback™ unit will be started according to instructions in the O&M manual (see Appendix B). Sampling will proceed once the unit has been operating normally for a period of at least one hour and the unit is determined to be operating properly with a product (permeate) flow rate of 1.1 gpm. This flow rate is the target-operating rate used by Gates Rubber Company.

Table 1. Test Objectives and Related Test Measurements for Evaluation of the Membralox® Silverback™ Model 900

Test	Test Objective	Test Measurement
1. Baseline: Normal soil loading rate.	Prepare a material balance for alkaline cleaner constituents and soils.	Processing rate of solution through unit. Chemical characteristics of feed solution. Chemical characteristics of recovered product. Volume and chemical characteristics of wastes removed from two-compartment tank. Quantity of concentrated cleaning solution added during testing. Chemical characteristics of concentrated cleaning solution.
	Evaluate the ability of the Silverback™ unit to process used alkaline cleaner solution and separate usable cleaner solution chemistry from bath contaminants.	Chemical characteristics of feed solution. Chemical characteristics of recovered product.
	Determine the alkaline cleaner recovery rate of the system, normalized based on production throughput and soil loading.	Volume of product produced. Production throughput for all associated cleaning baths. Chemical characteristics of feed solution. Chemical characteristics of recovered product.
	Determine labor requirements needed to operate and maintain the Silverback™ unit.	O&M labor required during test period.
	Determine the quantity of energy consumed by the Silverback™ unit during operation.	Quantity of energy used by pumps and filtration module.
	Determine the cost of operating the alkaline cleaning recovery system for the specific conditions encountered during testing.	Costs of O&M labor, materials, and energy required during test period. Quantity and price of fresh cleaning chemicals added during testing.
	Quantify/identify the environmental benefit.	Review historical waste disposal records and compare to current practices.
2. High Soil Loading Rate: Approx. two times the normal soil loading rate.	Same as baseline	Same as baseline

3.4.2 System Operation

The Silverback™ unit will be operated according to procedures contained in the USFilter O&M manual (see Appendix B). Sample collection and operating parameter measurements will be performed as indicated in Section 3.5 of this test plan. The unit will operate 24 hours/day for five days during each test run. The product flow rate will be maintained at approximately 1.1 gpm.[3] To maintain this rate, periodic adjustments will be made according to the instructions found in Appendix B.

The recirculation tank will not be discharged during a test run unless it is necessary to continue the operation of the unit. For example, this could occur if the processing rate could not be maintained at a minimum of 0.75 gpm due to solids buildup in the membrane.

At the completion of the first test cycle, the unit will be drained and cleaned and restarted following the same procedures as those used for set-up and system initialization.

The solution removed from the recirculation tank after the first test period will be stored in a tank located near the alkaline storage tank. To simulate a higher soil-loading rate during the second test period, one-tenth of the volume of the stored recirculation solution (about 10 gal.) will be transferred to the alkaline storage tank twice each day (at approximately 9 am and 5 pm). Whenever solution is transferred to the storage tank, the event will be recorded on the data collection form (i.e., time and volume transferred).

3.4.3 Sampling and Process Measurements

Sampling and process measurements will be taken according to the schedule presented in ***Table 2***. Sampling events and process measurements will be recorded on the form shown in ***Figure 3***. Each laboratory sample bottle will be labeled with the date, time, sample ID number, and test parameters required. Sample preparation methods are described in each individual analytical method.

Samples to be analyzed at an off-site laboratory will be accompanied by a chain of custody form. The samples will be transported in appropriate sample transport containers (e.g., coolers with packing and blue ice) by common carrier. The transport containers will be secured with tape to ensure sample integrity during the delivery process to the analytical laboratory. The Project Manager or designee will perform sampling, labeling, and ensure that samples are properly secured and transported to AMTest, Inc. in Redmond, WA, for analysis.

[3] The Model 900 is rated by the manufacturer to operate at a processing rate of 900 gpd, or approximately 0.75 gpm. The operating rate of 1.1 gpm was selected by Gates Rubber Company. The manufacturer of the unit indicated that it is not uncommon for this model to process solution at a higher rate than the design rate. Achievable processing rates depend on several factors including the chemistry and concentration of the cleaner and the characteristics and loading of the soil.

Table 2. Sampling Frequency and Parameters to be Measured

Sample Location	Frequency/Type	Parameters
Feed Sample Port	1/day (24-hr. composites)	alkalinity, phosphate, glycol, total phenol, total kjeldahl nitrogen (TKN), ammonia, oil and grease (OIL), total solids, and total suspended solids (TSS)
Silverback™ Unit (right side of two-compartment tank, where waste product accumulates)	1/week (grab at completion of test run)	alkalinity, phosphate, glycol, total phenol, total kjeldahl nitrogen (TKN), ammonia, oil and grease (OIL), total solids, and total suspended solids (TSS)
Silverback™ Unit (right side of two-compartment tank)	1/1st shift	temperature
Membrane Module Pressure Gauges	3/1st shift 1/2nd shift	membrane pressures (pump discharge/permeate)
Product Sample Port	1/day (24-hr. composites)	(alkalinity, phosphate, glycol, total phenol, total kjeldahl nitrogen (TKN), ammonia, oil and grease (OIL), total solids, and total suspended solids (TSS))
Product Line	3/1st shift 1/2nd shift	processing rate (instantaneous product flow and total product flow)
Concentrated cleaning solution	As needed.	volume added (gallons)
Concentrated cleaning solution	1 time during test	alkalinity, phosphate, glycol, total phenol, total kjeldahl nitrogen (TKN), ammonia, oil and grease (OIL), total solids, and total suspended solids (TSS)
Alkaline Cleaning Tank	1/1st shift	temperature
Product Storage Tank	1/1st shift	temperature

Sample ports have been installed to collect feed and product samples from the Silverback™ unit. To collect the composites (24-hr. composites), polyethylene tubes will be connected to the two sample ports and directed into 2.5 or five-gallon HDPE containers that are maintained at a cool temperature by placing the glass sample collection container in ice.

The samples will collect in the HDPE containers at a uniform rate (approximately 5 to 8 ml/min) through the 24-hour period (controlled by valve on sample ports). At the end of each 24 hr. sampling period, the HDPE will be labeled and properly stored.

Figure 3. Data Collection Form

Test Number: _______
Start Date: _______
Start Time: ________

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Date/ Time	Initials	Product Flow, gpm	Product Flow, (totalalizer) gal	Membrane Pressures		Temperature in Recirculation Tank, °F	Temperature in Recovered Product Tank, °F	Temperature in Soak Clean Tank, °F	**Sample No.**	**Sample Location**	**Notes and Observations**
				Pump Discharge, psi	Permeate, psi						

The waste product will be sampled from the recirculation tank, just prior to draining the unit. This sample will be collected while recirculation pump is running to assure that the contents of the recirculation tank are completely mixed. Fresh alkaline cleaning chemistry will be sampled from its storage container.

The temperature of the recirculation tank solution, soak clean tank solution, and recovered product tank solution will be measured using a thermometer (NSF certified). The membrane pressure readings will be taken from instrumentation integrated on the Silverback™ unit. The instantaneous flow rate and total flow of processed solution will also be taken from instrumentation integrated on the Silverback™ unit. The readings from the flow totalizer will be used to determine the quantity of solution processed by the unit. Due to the importance of this measurement, the accuracy of the flow totalizer will be determined by comparing the recorded flow rate to a manual “stopwatch and bucket” measurement. Based on these results, recorded values will be corrected, if necessary.

Electricity use will be calculated by determining the power requirements and cycle times of pumps and other powered devices. Gates Rubber Company will provide the cost of labor, electricity, and other items needed for a cost analysis.

3.4.4 Production Measurements

Gates Rubber Company routinely monitors processing volumes of each type of part (referred to as “group codes”). Also, they have available average surface area data for each group code (in^2) and type of metals processed. This information will be used to calculate the square footage of parts cleaned each day.

3.5 Analytical Procedures

3.5.1 Alkaline Cleaner Constituents

Alkaline cleaners used for metal cleaning are formulated from three separate chemical groups, some of which tend to overlap in function. These three groups are builders, surfactants, and additives.

Builders are inorganic metal salts that provide alkalinity to the cleaning solution. Typical builders include hydroxide, silicates (e.g., sodium metasilicate, sodium sesquisilicate, and sodium orthosilicate), phosphates (e.g., tetrasodium pyriphosphate, sodium tripolyphosphate, and sodium hexametaphosphate), sodium carbonate (i.e., soda ash), and sodium tetraborate (i.e., borax). During cleaning, the role of a builder/alkali is saponification of fatty oil, where insoluble fatty oil is converted to soluble glycerin and soluble soap. The process of saponification consumes alkali. [Ref. 1]

Surfactants (i.e., surface-active agents) are organic molecules that have two ends: a hydrophilic (water loving) end and a hydrophobic (water hating) end. Surfactants group together to form structures known as micelles, which are responsible for the solubilization of oil. The size of the surfactant micelle grows as it solubilizes oil and contaminants, creating an emulsion. During microfiltration, larger micelles (those which have solubilized oil) are retained on the concentrate side of the filter, while the smaller micelles pass through. [Ref. 1]

Additives are more vague in function than builders and surfactants. Often additives are used to promote the sequestration of metals in solution or aid in rust prevention. [Ref. 1]

The constituents of the PAVCO cleaner are (1) builders: sodium hydroxide and phosphate (2) surfactants: glycol ether and octylphenol, and (3) additive: amine.

To monitor for changes in builders, tests will be performed to measure alkalinity (EPA 310.1) and phosphates (EPA 200.7). To monitor for changes in surfactants, tests will be performed for glycol (GC-FID) and total phenol (EPA 420.2). Amine will be monitored by testing for total kjeldahl nitrogen (TKN) (EPA 351.3) and ammonia (EPA 350.1).

Regarding the method for analysis of glycol, the laboratory is working with samples to refine a matrix specific extraction and preparation procedure. Exact method specifics and details will be summarized by the laboratory and presented in the verification test report.

A summary of analytical tests is presented in ***Table 3***.

3.5.2 Oil

Oil is contributed to the cleaner bath when parts are processed. The oil is a combination of machining and cutting oils and coolants that are used in metalworking. These fluids may contain mineral oils, natural oils, fats and derivatives, or synthetic lubricants. Oil loading and the efficiency of oil separation will be measured by performing oil measurements on both the feed and product streams of the Silverback™ unit.

The analytical method for measuring oil that will be used is a modified organics and hydrocarbon gas chromatographic method, EPA Method 8015 (modified), the Northwest Total Petroleum Hydrocarbon – Extended Diesel (NWTPH-DX) method (See Appendix F). A gravimetric method for measuring both oil and grease in aqueous and sludge samples was not chosen for analytical testing. Concerns about possible interference's and "false positives" for oil concentration from surfactants and/or proprietary chemicals within the system under evaluation led to consider alternative analytical methods, or at least to demonstrate the efficacy of standard methods to these materials and sample matrices.

Table 3. Summary of Analytical Tests and Requirements

Parameter	Test Method	Sample Bottle	Sample Volume Required	Preservation / Handling	Hold Time
Tests applicable to tracking contaminants entering/removed from the alkaline cleaner:					
Oil	EPA 8015 modified (NWTPH-DX)	2.5 or 5-gal HDPE	150 ml	Acidify with 1 mL conc. HCl at lab, cool storage (<4^{o}C)	28 days
Total Solids	EPA Method 160.3	2.5 or 5-gal HDPE	150 ml	cool storage (<4^{o}C)	7 days
TSS	EPA Method 160.2	2.5 or 5-gal HDPE	same as total solids	cool storage (<4^{o}C)	7 days
Tests applicable to tracking constituents of the alkaline cleaner:					
Alkalinity	EPA 310.1	2.5 or 5-gal HDPE	500 ml	cool storage (<4^{o}C)	14 days
Phosphate	EPA 200.7	same bottle as oil	125 ml	Adjust to pH <2 at lab with conc. H_2SO_4	28 days
Glycol	GC-FID	2.5 or 5-gal HDPE	500 ml	cool storage (<4^{o}C)	28 days
Total Phenol	EPA 420.2	same bottle as oil	125 ml	cool storage (<4^{o}C), adjust to pH <2 at lab with conc. H_2SO_4	28 days
Amine: TKN	EPA 351.3	same bottle as oil	125 ml	cool storage (<4^{o}C), adjust to pH <2 at lab with conc. H_2SO_4	28 days
Amine: NH3	EPA 350.1	same bottle as oil	125 ml	cool storage (<4^{o}C), adjust to pH <2 at lab with conc. H_2SO_4	28 days

"Neat" samples of the exact formulated oil lubrication products, which are used on the parts themselves, will be characterized and used as calibration standards in these quantitative analyses (see appendix for chromatograms). Therefore, by using known dilutions of "neat" standards, calibration curves and reference solutions can be drawn (reference solutions were used for quantification purposes). EPA Method 8015 can then be used to quantify the oil in the cleaner separate from the PAVCO cleaner compounds. A copy of the method is attached for reference. (see Appendix F)

An initial characterization and evaluation of these "neat" formulated products using the modified Method 8015 was performed by the analytical laboratory (AMTest, Inc. of Redmond, WA). Modifications to the Method 8015 involved slight changes in the ramp time within the gas chromatographic program, which were within the proscribed acceptable method modifications. Each type of oil evaluated yielded a characteristic chromatographic signature. Based on the information received, no one particular oil product is known to dominate over the others. Using the aliquots from the neat solutions of the different formulated products, a mixed reference standard was created and a range of calibration concentration standards derived. Results are reported in milligrams/liter (mg/L) oil/grease. Therefore, by developing calibration curves for the oils used at Gates Rubber, EPA Method 8015 can quantify the oil in the cleaner separate from the cleaner compounds used at Gates Rubber. Preliminary samples were collected from Gates Rubber and analyzed. The analysis results verified the Method 8015 (modified) could successfully quantify the oils.

Another reference step evaluated the efficacy of the modified Method 8015 test method for these samples and matrices. Aliquots were analyzed using the modified Method 8015, the conventional freon extraction-gravimetric method, as well as the recently approved EPA Method 1664 (hexane extract). The modified Method 8015 and freon methods yielded comparable results. Although the freon method yielded acceptable results, freon has been phased out as an acceptable material under the Montreal Accord, and hence will not be in use within analytical methods in the very near future. The hexane extraction method did not yield successful extraction results. The aqueous matrix turned milky (akin to liquid gelatin), requiring several cleanup steps and resulting poor sample recovery (un-reproducibility of results). Due to the arduous sample preparation that would have been required, these test samples were not carried through to analyses. The cost for such analyses would be prohibitively expensive, so the hexane extraction method was discounted from further consideration in this study.

3.5.3 Total Solids and Suspended Solids

Solid material is contributed to the cleaning bath when parts are processed. These particles are known to impair the efficiency of the cleaner. The Silverback™ unit removes larger particles in the filter basket (25 µ) when solution first enters the unit, and it retains smaller particles in the recirculation tank during microfiltration (0.2 µ pore size).

To determine the effectiveness of the Silverback™ unit with regard to removal of particulates, tests for total filterable residue (EPA 160.2) will be performed. The referenced method produces values commonly referred to as total suspended solids (TSS). The total residue test (EPA 160.3) will be used in material balance calculations. The associated calculations are presented in Section 4.2 Data Reduction, Validation, and Reporting.

4.0 QUALITY ASSURANCE/QUALITY CONTROL REQUIREMENTS

Quality Assurance/Quality Control activities will be performed according to the applicable section of the Environmental Technology Verification Program Metal Finishing Technologies Quality Management Plan (ETV-MF QMP) [Ref. 3].

4.1 Quality Assurance Objectives

The first QA objective is to ensure that the process operating conditions and test methods are maintained and documented throughout each test and laboratory analysis of samples. The second QA objective is to use standard test methods (where possible) for laboratory analyses. The test methods to be used are listed in Table 3.

4.2 Data Reduction, Validation, and Reporting

4.2.1 Internal Quality Control Checks

Raw Data Handling. Raw data is generated and collected by laboratory analysts at the bench and/or sampling site. These include original observations, printouts, and readouts from equipment for sample, standard, and reference QC analyses. Data is collected both manually and electronically. At a minimum, the date, time, sample ID, instrument ID, analyst ID, raw signal or processed signal, and/or qualitative observations will be recorded. Comments to document unusual or non-standard observations also will be included on the forms, as necessary. The form presented in ***Figure 3*** will be used for recording data on-site. Additionally, process logs generated by Gates Rubber, an ISO 9001 certified company, will be copied to supplement these forms. These logs show, among other items, chemical additions and production throughput. These forms will be reviewed and copied on a daily basis.

The on-site Project Team member will generate Chain of custody forms and these forms will accompany samples when they are shipped off-site.

Raw data will be processed manually by the analyst, automatically by an electronic program, or electronically after being entered into a computer. The analyst will be responsible for scrutinizing the data according to laboratory precision, accuracy, and completeness policies. Raw data bench sheets and calculation or data summary sheets will be kept together for each sample batch. From the standard operating procedure and the raw data bench files, the steps leading to a final result may be traced. The ETV-MF Program Manager will maintain process-operating data for use in verification report preparation.

Data Package Validation. The generating analyst will assemble a preliminary data package, which shall be initialed and dated. This package shall contain all QC and raw data results, calculations, electronic printouts, conclusions, and laboratory sample tracking information.

A second analyst will review the entire package and check sample and storage logs, standard logs, calibration logs, and other files, as necessary, to ensure that all tracking, sample treatments, and calculations are correct. After the package is reviewed in this manner, a preliminary data report will be prepared, initialed, and dated. The entire package and final report will be submitted to the Laboratory Manager.

The Laboratory Manager shall be ultimately responsible for all final data released from the laboratory. The Laboratory Manager or designee will review the final results for adequacy to task QA objectives. If the manager or designee suspects an anomaly or non-concurrence with expected or historical performance values, or with task objectives for test specimen performance, the raw data will be reviewed, and the generating and reviewing analysts queried. If suspicion about data validity still exists after internal review of laboratory records, the manager will authorize a re-test. If sufficient sample is not available for re-testing, a re-sampling shall occur. If the sampling window has passed, or re-sampling is not possible, the manager will flag the data as suspect. The Laboratory Manager signs and dates the final data package.

Data Reporting. A report signed and dated by the Laboratory Manager will be submitted to the ETV-MF Project Manager. The ETV-MF Project Manager will decide the appropriateness of the data for the particular application. The final report contains the laboratory sample ID, date reported, date analyzed, the analyst, the SOP used for each parameter, the process or sampling point identification, the final result, and the units. The ETV-MF Program Manager shall retain the data packages as required by the ETV-MF QMP [Ref. 3].

4.2.2 Calculation of Data Quality Indicators

Analytical performance requirements are expressed in terms of precision, accuracy, representativeness, comparability, completeness, and sensitivity (PARCCS). Summarized below are definitions and QA objectives for each PARCCS parameter. Duplicates and spike duplicates will be performed on one out of every ten samples. Sample splitting will occur in the analytical laboratory.

Matrix spike/spike duplicates will be performed at a frequency of 10% (one out of every ten samples), as specified in the modified EPA 8015 method protocol. Additional spikes and duplicates will be performed where applicable, with respect to the analyte of interest. Please refer to the methods for reference.

With respect to programmatic duplicates, field splits (duplicates) of pre-treatment (and post-treatment) samples will be performed at a frequency of one per field sample batch processed and/or ten field samples of a given type (e.g., at least one per ten days of field samples), within the prescribed holding times and method requirements.

4.2.2.1 Precision

Precision is a measure of the agreement or repeatability of a set of replicate results obtained from duplicate analyses made under identical conditions. Precision is estimated from analytical data and cannot be measured directly.

The precision of a duplicate determination can be expressed as the relative percent difference (RPD), and calculated as:

$$\text{RPD} = \{(|X_1 - X_2|)/(X_1 + X_2)/2\} \text{ x } 100 = \left\{ \frac{|X_1 - X_2|}{\frac{(X_1 + X_2)}{2}} \right\} \text{x } 100$$

where, X_1 = larger of the two observed values and X_2 = smaller of the two observed values.

Multiple determinations will be performed for each test on the same test specimen. The replicate analyses must agree within the relative percent deviation limits provided in ***Table 4.***

4.2.2.2 Accuracy

Accuracy is a measure of the agreement between an experimental determination and the true value of the parameter being measured. Accuracy is estimated through the use of known reference materials or matrix spikes. It is calculated from analytical data and is not measured directly. Spiking of reference materials into a sample matrix is the preferred technique because it provides a measure of the matrix effects on analytical accuracy. Accuracy, defined as percent recovery (P), is calculated as:

$$P = \left[\frac{(SSR - SR)}{SA} \right] \text{x } 100$$

where: SSR = spiked sample result

SR = sample result (native)

SA = the concentration added to the spiked sample

Analyses will be performed with periodic calibration checks with traceable standards to verify instrumental accuracy. These checks will be performed according to established procedures in the contracted laboratory(s) that have been acquired for the U.S. Filter verification test. Analysis with spiked samples will be performed to determine percent recoveries as a means of checking method accuracy. QA objectives will be satisfied if the *average* recovery is within the goals described in ***Table 4***.

Table 4. QA Objectives

Critical Measurements	Matrix	Method	Reporting Units	Method of Determination	MDL	Precision (RPD)	Accuracy (% Recovery)	Completeness
Oil	Water	EPA Method 8015 modified	mg/l	GC-FID	0.2	≤ 30	50 - 150	95
Total Solids	Water	EPA Method 160.3	mg/l	Gravimetric	1.0	≤ 30	80 - 120	95
TSS	Water	EPA Method 160.2	mg/l	Gravimetric	1.0	≤ 30	80 - 120	95
Alkalinity	Water	EPA 310.1	mg/l	Titration	1.0	≤30	8-120	95
Phosphate	Water	EPA 200.7	mg/l		0.005	≤30	8-120	95
Glycol	Water	**#1**	mg/l	GC-FID		≤30	8-120	95
Total Phenol	Water	EPA 420.2	mg/l		0.005	≤30	8-120	95
TKN	Water	EPA 351.3	mg/l		0.25	≤30	8-120	95
NH3	Water	EPA 350.1	mg/l		0.005	≤30	8-120	95
Temperature	Water	Thermometer	^oC (^oF)	-	-	-	1	100
Chemical additions	Water		gallons	-	-	-		-
Flow	Water	Flow meter (totalizer)	gallons	-	-	-	**#2**	-

1. Glycol will be determined using a matrix specific GC-FID analysis (no applicable method number). Exact method specifics and details will be presented in the verification testing report.
2. Accuracy will be verified by stopwatch and bucket method.

4.2.2.3 Completeness

Completeness is defined as the percentage of measurements judged to be valid compared to the total number of measurements made for a specific sample matrix and analysis. Completeness is calculated using the following formula:

$$\text{Completeness} = \frac{\text{Valid Measurements}}{\text{Total Measurements}} \times 100$$

Experience on similar projects has shown that laboratories typically achieve about 90 percent completeness. QA objectives will be satisfied if the percent completeness 90 percent or greater as specified in ***Table 4***.

4.2.2.4 Comparability

Comparability is another qualitative measure designed to express the confidence with which one data set may be compared to another. Sample collection and handling techniques, sample matrix type, and analytical method all affect comparability. Comparability is limited by the other PARCCS parameters because data sets can be compared with confidence only when precision and accuracy are known. Comparability will be achieved in the U.S. Filter technology verification by the use of consistent methods during sampling and analysis and by traceability of standards to a reliable source.

4.2.2.5. Representativeness

Representativeness refers to the degree to which the data accurately and precisely represents the conditions or characteristics of the parameter represented by the data. For the purposes of this demonstration, representativeness will be achieved by presenting identical analyte samples to the specified lab(s) and executing consistent sample collection and mixing procedures.

4.2.2.6. Sensitivity

Sensitivity is the measure of the concentration at which an analytical method can positively identify and report analytical results. The sensitivity of a given method is commonly referred to as the detection limit. Although there is no single definition of this term, the following terms and definition of detection will be used for this program.

Instrument Detection Limit (IDL) is the minimum concentration that can be measured from instrument background noise

4.2.2.7 Method Detection Limit

(MDL) is a statistically determined concentration. It is the minimum concentration of an analyte that can be measured and reported with 99 percent confidence that the analyte concentration is greater than zero as determined in the same or a similar matrix [because of the lack of information on analytical precision at this level, sample results greater than the MDL but less than the practical quantification limit (PQL) will be laboratory qualified as “estimated”]

MDL is defined as follows for all measurements:

$$MDL = t_{(n-1,1-\alpha = 0.99)} \times s$$

where: MDL = method detection limit

s = standard deviation of the replicate analyses

$t_{(n-1,1-\alpha = 0.99)}$ = students t-value for a one-sided 99% confidence level and a standard deviation estimate with n-1 degrees of freedom.

Method Reporting Limit (MRL) is the concentration of the target analyte that the laboratory has demonstrated the ability to measure within specified limits of precision and accuracy during routine laboratory operating conditions [This value is variable and highly matrix dependent. It is the minimum concentration that will be reported without qualifications by the laboratory].

4.2.2.8 Cleaner Recovery Efficiency

The recovery efficiencies are calculated based on mass balances of the dissolved species. These calculations are performed for each daily set of paired analytical results. The equation for alkalinity recovery calculation is shown below. Volumes collected in units of gallons will be mathematically converted to liters prior to calculation. Other recovery efficiency equations (i.e., phosphate, glycol ether, octyphenol, and amine) will follow the same format.

$$A_{eff}\ (\%) = [(A_{prod} \times Prod_{vol}) / (A_{feed} \times Feed_{vol})] \times 100\%$$

where: A_{eff} = alkalinity recovery efficiency

A_{prod} = product stream alkalinity concentration (grams/liter)

$Prod_{vol}$ = product volume collected during the cycle (liters)

A_{feed} = feed solution alkalinity concentration (grams/liter)

$Feed_{vol}$ = feed solution volume processed during the cycle (liters)

4.2.2.9 Mass Balance

The equation for mass balance uses the equation above and adds a term for the alkalinity contained in the waste stream so that the mass balance for the alkalinity is shown below. Volumes collected in units of gallons will be mathematically converted to liters prior to calculation. Other mass balance equations will be similar.

$$\text{mass bal. } (\%) = [(A_{prod} \times Prod_{vol}) + (A_{waste} \times Waste_{vol}) / (A_{feed} \times Feed_{vol})] \times 100\%$$

where: A_{waste} = waste stream alkalinity concentration (grams/liter)

$Waste_{vol}$ = waste stream volume (liters)

4.2.2.10 Energy Use

Energy requirements for the Silverback™ unit will be calculated by summing the total quantity of horsepower hours and dividing by 1.341 HP-hr/kWh to arrive at electricity needs. Volumes collected in gallons will be mathematically converted to liters prior to calculation.

Heat required to raise solution temperature

$$\text{(BTUs/cycle)} \qquad = \text{Prod}_{vol} \text{ x } 2.2\ \frac{lb}{L} \text{ x } \Delta T \text{ x } 1\ \frac{BTU}{lb°F}$$

Where: Prod_{vol} = product volume collected during the cycle (liters)

ΔT = average temperature difference between recovered product tank and soak clean tank (F°)

4.2.2.11 Cost Analysis

This analysis will quantify the cost benefit of the technology. The costs for operating the Silverback™ unit at Gates Rubber Company will be calculated to operating costs for a time period prior to installation of the unit at Gates Rubber Company. For the baseline conditions, the most recent applicable data available from Gates Rubber Company will be used. The cost analysis will compare operating costs, including costs for: alkaline cleaner solution, drag-out losses, other materials, waste treatment/disposal, labor, and utilities.

4.2.2.12 Waste Generation Analysis

This analysis will quantify the environmental benefit of the technology. The waste generation rates for operating the alkaline cleaner system with the Silverback™ unit at Gates Rubber Company will be calculated and compared to waste generation rates for a time period to installation of the unit at Gates Rubber Company. For the baseline conditions, the most recent applicable data from Gates Rubber Company will be used. The waste generation analysis will consider type/characteristics of wastes generated and volume and frequency of waste generated.

4.3 Quality Audits

Technical System Audits. An audit will be performed during verification testing by the *CTC* QA Manager according to Section 2.9.3 Technical Assessments of the ETV-MF QMP [Ref. 3] to ensure testing and data collection are performed according to the test plan requirements. In addition to the *CTC* Technical System Audit, the EPA Quality Assurance Manager may also conduct an audit to assess the quality of the verification test.

Internal Audits. In addition to the internal laboratory quality control checks, internal quality audits will be conducted to ensure compliance with written procedures and standard protocols.

Corrective Action. Corrective Action for any deviations to established quality assurance and quality control procedures during verification testing will be performed according to section 2.10 Quality Improvement of the ETV-MF QMP [Ref. 3].

Laboratory Corrective Action. Examples of non-conformances include invalid calibration data, inadvertent failure to perform method specific QA, process control data outside specified control limits, failed precision and/or accuracy indicators, etc. Such non-conformances will be documented on a standard laboratory form. Corrective action will involve taking all necessary steps to restore a measuring system to proper working order and summarizing the corrective action and results of subsequent system verifications on a standard laboratory form. Some non-conformances are detected while analysis or sample processing is in progress and can be rectified in real time at the bench level. Others may be detected only after a processing trial and/or sample analyses are completed. Typically, these types of non-conformances are detected by the Laboratory Manager. In all cases of non-conformance, sample re-analysis will be considered as one source of corrective action by the Laboratory Manager. If insufficient sample is available or the holding time has been exceeded, complete re-processing may be ordered to generate new samples if a determination is made by the Task Leader that the non-conformance jeopardizes the integrity of the conclusions to be drawn from the data. In all cases, a non-conformance will be rectified before sample processing and analysis continues.

5.0 PROJECT MANAGEMENT

5.1 Organization/Personnel Responsibilities

The ETV-MF Project Team that is headed by *CTC* will conduct the evaluation of the Membralox® Silverback™ Model 900. The ETV-MF Program Manager, Donn Brown, will have ultimate responsibility for all aspects of the technology evaluation. The ETV-MF Project Team Leader assigned to this evaluation is George Cushnie. Mr. Cushnie and/or his staff member will be on-site throughout the test period and will conduct or oversee all sampling and related measurements.[4]

USFilter will be responsible for training assigned personnel on operation of the technology and will assist in startup of the system if necessary. They will also be on-call during the test period for response in the event of equipment problems.

Gates Rubber Company personnel will be responsible for operation of the Silverback™ equipment, related cleaning lines, and ancillary equipment such as

[4] The *CTC* ETV-MF Program Manager, Donn Brown, will make a determination as to the qualifications of any staff member assigned to the project. This will occur prior to testing.

pumps and system instrumentation. With instruction from the ETV-MF Project Team Leader or staff member, periodically Gates Rubber Company personnel will collect samples or record data from instrumentation readings. Gates Rubber Company personnel will also provide safety training as described in Section 9.0. The ETV-MF Team Leader and the Gates Rubber Company have the authority to stop work when unsafe or unacceptable quality conditions arise.

AMTest Laboratories is responsible for analyzing verification test samples. The Laboratory Manager, Kathy Fugiel, will be point of contact. AMTest Laboratories is accredited by the State of Washington, Department of Ecology for the analyses identified in this test plan.

The ETV-MF Project Manager and Gates Rubber have the authority to stop work when unsafe or unacceptable quality conditions arise. The *CTC* ETV-MF Program Manager will provide periodic assessments of verification testing to the EPA ETV Pilot Manager.

6.0 EQUIPMENT AND UTILITY REQUIREMENTS

All equipment and utilities required for this ETV-MF project are owned by and are currently installed at Gates Rubber Company. These utility requirements include:

- Electrical supply to panel: 560VAC, 60 Hz, three-phase
- Instrument air: <1 scfm, 80 psi, dry, oil-free
- Steam: 6 lb./hr., or
- Hot Water: 160^{o}F

7.0 HEALTH AND SAFETY PLAN

This Health and Safety Plan provides guidelines for recognizing, evaluating, and controlling health and physical hazards throughout the workplace. More specifically, the Plan specifies for assigned personnel, the training, materials, and equipment necessary to protect themselves from hazards created by acids, and any waste generated by the process.

7.1 Hazard Communication

All personnel assigned to the project will be provided with the potential hazards, signs and symptoms of exposure, methods or materials to prevent exposures, and procedures to follow, if there is contact with a particular substance. The Gates Rubber Company Hazard Communication Program will be reviewed during training and will be reinforced throughout the test period. All appropriate MSDS forms will be available for chemical solutions used during testing.

7.2 Emergency Response Plan

Gates Rubber Company has a contingency plan to protect employees, assigned project personnel, and visitors in the event of an emergency at the facility. This plan will be used throughout the project. All assigned personnel will be provided with information about the plan during training.

7.3 Hazard Controls Including Personal Protective Equipment

All assigned project personnel will be provided with appropriate personal protective equipment (PPE) and any training needed for its proper use, considering their assigned tasks. The use of PPE will be covered during training as indicated in Section 9.0.

The following PPE will be required and must be worn at all times while in the Gates Rubber facility: Steel toed footwear and eyeglasses with side splashguards.

The alkaline recycling system is essentially a closed-loop process and fully contained within the manufacturing building. There are no apparent hazards to the surrounding community due to operation or testing of the system.

7.4 Lockout/Tagout Program

No new equipment will be installed; therefore, a lockout/tagout program will not be necessary.

7.5 Material Storage

Any materials used during the project will be kept in proper containers and labeled according to Federal and State law. Proper storage of the materials will be maintained based on associated hazards. Spill trays or similar devices will be used as needed to prevent material loss to the surrounding area.

7.6 Safe Handling Procedures

All chemicals and wastes or samples will be transported on-site in non-breakable containers used to prevent spills. Spill kits will be strategically located in the project area. These kits contain various sizes and types of sorbents for emergency spill clean up. Emergency spill clean up will be performed according to the Emergency Response Plan.

8.0 WASTE MANAGEMENT

The alkaline recycling equipment will be tested on processes already in-place and operating at Gates Rubber Company. This equipment currently generates a waste as a result of weekly or biweekly discharging of the recirculation tank. This waste material is presently treated on-site and discharged to the local POTW, in accordance with local, state, and Federal laws.

During testing, no additional wastes will be generated other than the normal discharge of the recirculation tank. Gates Rubber Company using their normal practices will handle this waste. Therefore, no special or additional provisions for waste management will be necessary.

9.0 TRAINING

It is important that the verification activities performed by the ETV-MF Program be conducted with high quality and with regard to the health and safety of the workers and the environment. By identifying the quality requirements, worker safety and health, and environmental issues associated with each verification test, the qualifications or training required for personnel involved can be identified. Training requirements will be identified using the Job Training Analysis (JTA) Plan [Ref. 5].

The purpose of this JTA Plan is to outline the overall procedures for identifying the hazards and quality issues and training needs for each verification test project. This JTA Plan establishes guidelines for creating a work atmosphere that meets the quality, environmental, and safety objectives of the ETV-MF Pilot. The JTA Plan describes the method for studying ETV-MF project activity and identifying training needs. The ETV-MF Operation Planning Checklist (***Appendix C***) will be used as a guideline for identifying potential hazards, and the Job Training Analysis Form (***Appendix D***) will be used to identify training requirements. After completion of the form, applicable training will be performed. Training will be documented on the ETV-MF Project Training Attendance Form (***Appendix E***). Health and safety training will be coordinated with Jerry Capps, the Gates Rubber Company Human Resources Director.

10.0 REFERENCES

1. Peterson, David, "*Practical Guide to Industrial Metal Cleaning*," Hanser Gardner Publications, Cincinnati, OH, 1997.
2. Cushnie, George C., "*Pollution Prevention and Control Technology for the Electroplating Industry*," NCMS, 1994.
3. Concurrent Technologies Corporation, "*Environmental Technology Verification Program Metal Finishing Technologies (ETV-MF) Quality Management Plan.*" December 9, 1998.
4. US EPA Office of Research and Development,. "*Preparation Aids for the Development of Category IV Quality Assurance Project Plans,*" EPA/600/8-91/006, February, 1991.
5. Concurrent Technologies Corporation, "*Environmental Technology Verification Program Metal Finishing Technologies (ETV-MF) Pollution Prevention Technologies Pilot Job Training Analysis Plan,*" May 10, 1999.

11.0 DISTRIBUTION

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APPENDIX A

PAVCO CLEAN-R-120GR Material Safety Data Sheet

APPENDIX B

USFilter Membralox® Silverback™ Model 900 Operation and Maintenance Manual (selected sections)

Appendix C

ETV-MF Operation Planning Checklist

ETV-MF Operation Planning Checklist

The ETV-MF Project Manager prior to initiation of verification testing must complete this form. If a "yes" is checked for any items below, an action must be specified to resolve the concern on the Job Training Analysis Form.

Project Name: ____ Expected Start Date: _______________

ETV-MF Project Manager: ___________________________________

Will the operation or activity involve the following:	Yes	No	Initials & Date Completed
Equipment requiring specific, multiple steps for controlled shutdown? (e.g. in case of emergency, does equipment require more than simply pressing a "Stop" button to shut off power?) *Special Procedures for emergency shut-down must be documented in Test Plan.*			
Equipment requiring special fire prevention precautions? (e.g. Class D fire extinguishers)			
Modifications to or impairment of building fire alarms, smoke detectors, sprinklers or other fire protection or suppression systems?			
Equipment lockout/tagout or potential for dangerous energy release? *Lockout/tagout requirements must be documented in Test Plan.*			
Working in or near confined spaces (e.g., tanks, floor pits) or in cramped quarters?			
Personal protection from heat, cold, chemical splashes, abrasions, etc.? *Use Personal Protective Equipment Program specified in Test Plan.*			
Airborne dusts, mists, vapors and/or fumes? *Air monitoring, respiratory protection, and /or medical surveillance may be needed.*			
Noise levels greater than 80 decibels? *Noise surveys are required. Hearing protection and associated medical surveillance may be necessary.*			
X-rays or radiation sources? *Notification to the state and exposure monitoring may be necessary.*			
Welding, arc/torch cutting, or other operations that generate flames and/or sparks outside of designated weld areas? *Follow Hot Work Permit Procedures identified in Test Plan.*			
The use of hazardous chemicals? *Follow Hazard Communication Program, MSDS Review for Products Containing Hazardous Chemicals. Special training on handling hazardous chemicals and spill clean-up may be needed. Spill containment or local ventilation may be necessary.*			
Working at a height of six feet or greater?			

ETV-MF OPERATION PLANNING CHECKLIST

The ETV-MF Project Manager prior to initiation of verification testing must complete this form. If a "yes" is checked for any items below, an action must be specified to resolve the concern on the Job Training Analysis Form.

Project Name: ______________________________

ETV-MF Project Manager: ______________________________

Will the operation or activity involve the following:	**Yes**	**No**	**Initials & Date Completed**
Processing or recycling of hazardous wastes? *Special permitting may be required.*			
Generation or handling of waste?			
Work to be conducted before 7:00 a.m., after 6:00 p.m. and/or on weekends? *Two people must always be in the work area together.*			
Contractors working in *CTC* facilities? *Follow Hazard Communication Program.*			
Potential discharge of wastewater pollutants?			
EHS aspects/impacts and legal and other requirements identified?			
Contaminants exhausted either to the environment or into buildings? *Special permitting or air pollution control devices may be necessary.*			
Any other hazards not identified above? (e.g. lasers, robots, syringes) *Please indicate with an attached list.*			

The undersigned responsible party certifies that all applicable concerns have been indicated in the "yes" column, necessary procedures will be developed, and applicable personnel will receive required training. As each concern is addressed, the ETV-MF Project Manager will initial and date the "initials & date completed" column above.

ETV-MF Project Manager: ____________________ ____________________ ____________

(Name) (Signature) (Date)

APPENDIX D

Job Training Analysis Form

Job Training Analysis Form

ETV-MF Project Name: ____

Basic Job Step	**POTENTIAL EHS ISSUES**	**Potential Quality Issues**	**Training**

ETV-MF Project Manager:____
Name **Signature**

____
Date

Appendix E

ETV-MF Project Training Attendance Form

ETV-MF Project Training Attendance Form

ETV-MF Pilot Project:

Date Training Completed	Employee Name Last First	Training Topic	Test Score (If applic.)

ETV-MF Project Manager: ____

APPENDIX F

Oil Analysis

EPA Method 8015, Modified
(Northwest Total Petroleum Hydrocarbon – Extended Diesel) (NWTPH-DX)

NWTPH-DX
Diesel Range Organics In Soil And Water

Summary

The NWTPH-D Method adapts EPA SW-846 Methods 3540 and 8000 and covers the quantitative analysis of semi-volatile petroleum products in soils. The method involves extracting the sample with methylene chloride and injecting of a portion of the extract into a gas chromatograph equipped with a flame ionization detector. This method specifies criteria for the identification and quantitation of semi-volatile petroleum products. When the type of fuel is unknown #2 diesel will be used to quantitate the sample. The reporting limit is 25 mg/kg for soil and 0.10 mg/l for water samples eluting from the jet fuels range to the diesel #2 range. For petroleum products eluting after diesel #2 the reporting limits are 100 mg/kg for soil and 0.20 mg/l for water (assuming 100% total solids for soil).

Equipment and Reagents

Gas Chromatograph
Flame Ionization detector
Column: J & W DB-5 30M x . 32mm with .25um film thickness
Capillary column
Maxima Data System
Analytical Balance accurate to at least 0.001g
Horn Sonicator
Volumetric Flasks, 10ml Ground Glass Stoppered
150ml beakers
Sodium Sulfate
Methylene Chloride
K-D Equipment (refer to K-D section)
Nitrogen evaporator
Sulfuric Acid, concentrated
Silica gel cartridges
Various Petroleum products for standards

Collection Requirements

All samples should be collected in I-Chem containers and preserved at 4 degrees Celsius until extracted. The holding time from the date of collection to extraction, is 14 days for soils and preserved water. For unpreserved water, the holding time is 7 days. Preservation is accomplished by adjusting pH to about 2 using 1:1 HC1.

Standards

<u>Fuel Stock Standard</u>:
Choose the appropriate fuel for comparison to the sample fingerprint. Weight approximately 0.10 g into a 10ml vol. flask and dilute to volume with DCM. Label and record the exact concentration.

Calculate the concentration as follows:

$$\text{Stock Conc. ug/ml} = \frac{\text{weight diesel (g)} \times 1{,}000{,}000 \text{ ug/g}}{10\text{ml}}$$

Calibration Standard:
Prepare calibration standards from the stock diesel standard at concentrations of 25, 50, 200, 200, 300 ug/ml by adding appropriate volumes to a 10ml vol. flask and diluting to volume with methylene chloride. For fuels heavier than diesel #2, prepare standards at concentrations of 50, 100, 150, 300, and 400 ug/ml.

To calculate volume (ul) of stock standard to add to 10ml vol. flask use the equation below:

$$\text{Vol. Diesel Stock, ul} = \frac{\text{Cal. Std. Conc ug/ml} \times 1000\text{ug} \times 10}{\text{Diesel stock ug/ml}}$$

Dilute the flask to 10mls with DCM.

Stock Surrogate Standard:
Make up a surrogate of bromofluorobenzene and 2-fluorobiphenyl, which contains approximately 8000 ug/ml by weighing about .080 g of the surrogate compounds into a 10ml volumetric flask and filling to volume with mehtylene chloride.

Working Surrogate Spike (800 ug/ml):
Add the appropriate volume of the stock standard to a 10ml volumetric flask which has been filled with 5mls of methylene chloride taking care not to add the surrogate standard solution into the solvent without contacting the neck of the flask. Fill the flask to volume, stopper and mix. Store at 4 degrees Celsius.

$$\text{Volume Surrogate Stock, ul} = \frac{800 \text{ ug/ml} \times 10\text{ml} \times 1000\text{ul}}{\text{Surrogate stock conc. ug/ml}}$$

GC/FID PARAMETERS FOR FUEL SCANS

Instrument Parameters:

Column:	J & W DB-5 30M x .32mm with .25 film thickness, capillary column
Injection Sample Volume:	2 ul
Injector Temperature:	290 C
Ion Block Temperature:	300 C
Initial Temperature:	35 C
Initial Time:	5 minutes
Initial Rate:	10 degrees/min.
Final Temperature:	300 C
Final Time:	5 minutes
Purge Valve on time:	1.5 minutes
Purge Valve off time:	36 minutes

Purge Valve on time:	1.5 minutes
Purge Valve off time:	36 minutes
Hydrogen Flow:	25 – 30 ml/min.
Air Flow:	300 – 400 ml/min.
Make-up Gas Flow:	30 ml/min.
Carrier Gas:	Helium

Helium Carrier gas Head Pressure = 12 psi

Sample Extraction Soil

Accurately weigh approximately 20 grams of soil (note that if the sample is hydrated, more than 20 grams is needed) 20 grams of anhydrous sodium sulfate into a 150ml beaker and mix completely with a spatula. The mixture should have a grainy texture. If it forms a clump, add more sodium sulfate, grind to a grainy texture and note this in the extraction log. Add 100 ul of Working Surrogate spike and 50 ml of methylene chloride; sonicate this for 3 min. utilizing the horn sonicator. (Refer to Horn sonicator instructions if unfamiliar with the operation of the instrument at the end of this SOP).

Allow the mixture to stand. Collect the extract in a 250 ml Kuderna-Danish (KD) Flask to which is connected a ten ml. concentrator tube and a sodium sulfate drying apparatus.

Repeat the extraction twice more using 50ml of Methylene Chloride and add the extract to the same KD flask. Attach a 3 ball Snyder column and concentrate the extract to a final volume of 10 ml. If the extract is highly colored or forms a precipitate, a dilution may be necessary to stay within the calibration range. If samples need to be cleaned up, refer to “Clean-up” procedure at the end of this method.

Store the samples at 4 degrees Celsius in a glass vial until ready for analysis.

Water Extraction Procedure

Pour 500 mls of the sample into a 2 liter sepratory funnel. Adjust the pH to 2 if needed. Add 200 ul of surrogate working std. Extract the sample with 50 mls of DCM. Pour the extract through sodium sulfate into a K-D set up. Extract the sample twice more with 50 mls. DCM adding the extract to the K-D set up. Concentrate the sample to 5 – 10 mls on a steam bath. Remove the ampule and continue to concentrate on a N-Vap to below 2 mls. Adjust the final volume to 2 mls in a vol. flask. Clean-up the sample if needed using the procedure at the end of the SOP.

Determine the Total Solids Percent of soil sample.

<u>GC Run to include the following:</u>

1. Five point calibration curve
2. 10% duplicates
3. Surrogate std (100 ug/L working surrogate spike to 10 ml)
4. Mid std check every ten samples analyzed
5. End std check at the end of each run.

Data Validation:

1. Continuing calibration checks and end checks must fall +/- 15% of the known value of the std.
2. Surrogate recoveries must be between 50% - 150%.
3. Standard curve must have a minimum correlation of 0.99.

Sample Calculations

The retention time range windows for integration must be adjusted to incorporate the majority of the components of the petroleum product of interest. If an exact match cannot be made, a standard is chosen which closely represents the sample. In all cases, the selected retention time window used for quantitation must, at a minimum, include any unresolved envelope of compounds as well as all discrete components peaks with an area greater than or equal to 10% of the largest peak. These must be integrated to the baseline as a group.

Be sure to subtract the area of the surrogates if the surrogate falls within the retention time window.

Adjustments of retention time windows may be made if interferences are present, i.e, overlap of oil into diesel area.

$$\text{Sample Conc. mg/kg} = \frac{\text{Sample conc. ug/ml} \times v \times DF}{\text{Sample weight} \times TS}$$

V	=	Final Volume of extract
DF	=	Dilution Factor
TS	=	Decimal percent solids of sample

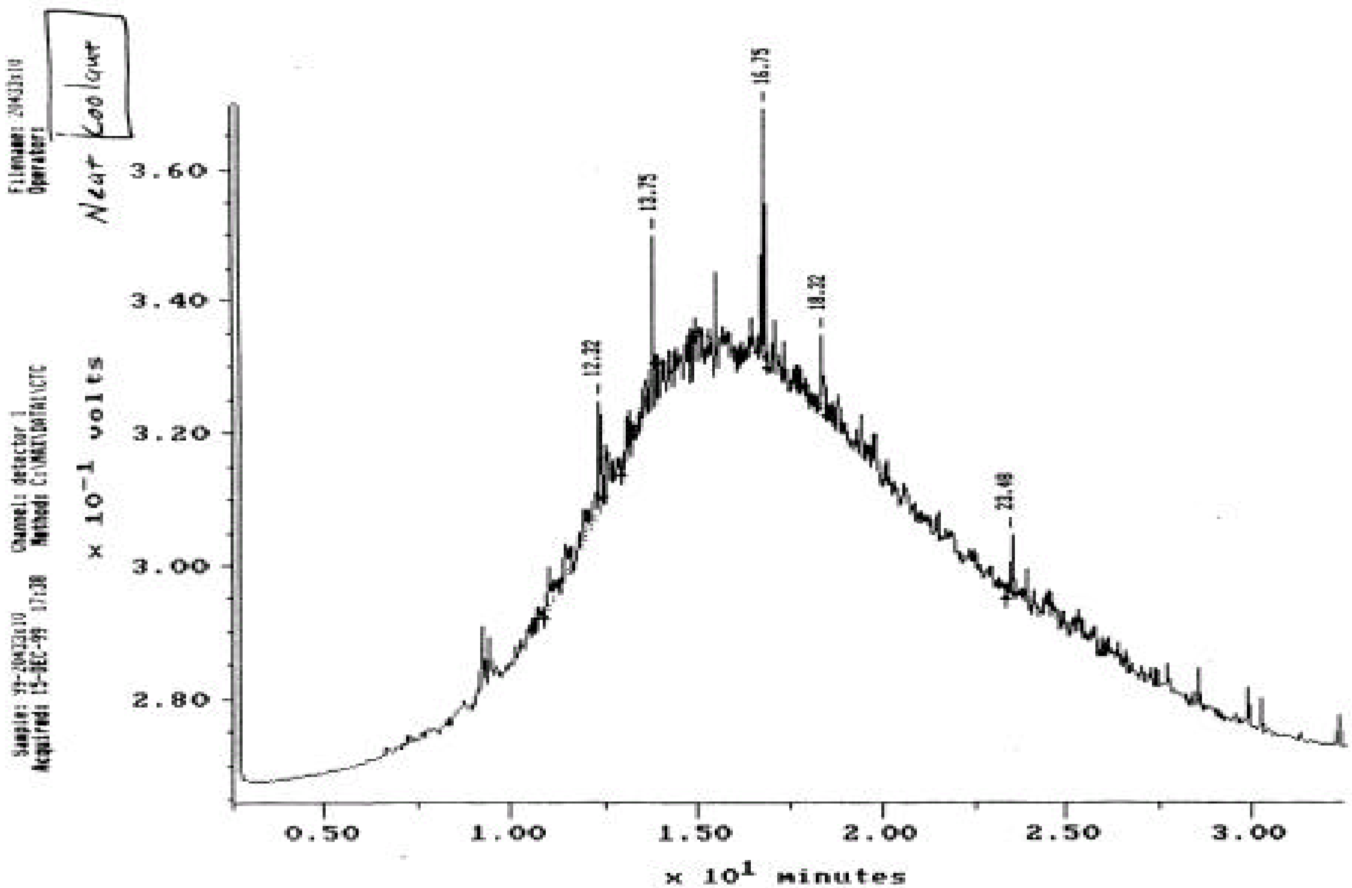
Horn Sonicator Settings

Sonicator Type:	Ultrasonic, Inc. Model W-385 (475 watt) with No. 207 ¾" Tapped Disruptor Horn
Settings:	3 minutes Output Control Knob: 10 Mode: Pulse Percent Duty Knob: 50%

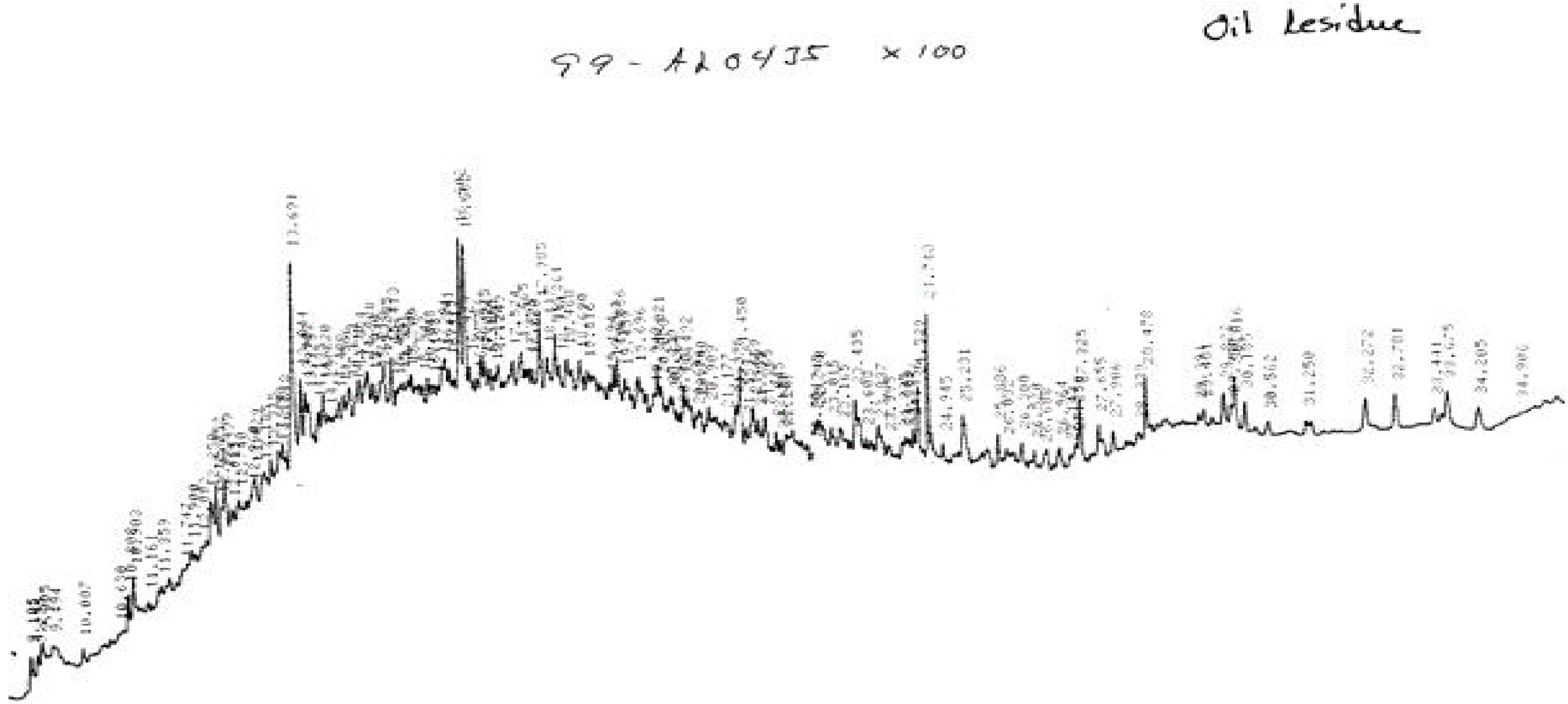
Sample Clean-up Procedure:

When samples contain a significant amount of naturally occurring non-petroleum organics, e.g. leaf litter, bark, etc. which may contribute to biogenic interference, the following clean-up technique may be employed to assist in their reduction or elimination.

1. Transfer 2ml of the sample extract to a 4 ml vial.
2. Add .3 to .5 ml concentrated sulfuric acid to the vial and shake for 1 minute.
3. Allow the phases to separate and transfer the upper layer to another 4 ml vial.
4. Add about .4g of silica to the vial and shake.
5. Repeat the procedure a second time. Transfer the cleaned extract to an auto sampler vial for analysis.
6. If the clean-up affects the analyte of interest, clean the standards in the same way as the samples.



16.75
13.75
18.32
12.32
23.48
3.60
3.40
3.20
3.00
2.80
x 10-1 volts
0.50
1.00
1.50
2.00
2.50
3.00
x 101 minutes


99 - A20435 x 100
Oil Residue